# PATENT SPECIFICATION

DRAWINGS ATTACHED

**849,929**

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COMPLETE SPECIFICATION

## Improved Method and Apparatus for Catalyst Activation

We, PHILLIPS PETROLEUM COMPANY, a corporation organised under the laws of the State of Delaware, United States of America, of Bartlesville, Oklahoma, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process and apparatus for activating solid catalyst and more particularly to the activation of particulate metallic oxide catalysts in a fluidized bed. In another of its more specific aspects it relates to an improved process for activating a particulate polymerization catalyst comprising chromium oxide, a portion of which is hexavalent.

Many solid catalyst compositions, particularly those employed in hydrocarbon conversion operations such as polymerization, cracking and dehydrogenation require, for maximum effectiveness, an activation treatment which comprises subjecting the raw catalyst to elevated temperatures for an interval of time, while passing over this catalyst a stream of gas which is inert, non-oxidizing, non-reducing, or dry, depending on the particular nature of the catalyst and its intended use. In the activation of particulate metallic oxide catalysts, especially those used in the polymerization art, it is often advantageous to heat the catalyst by contacting it while in a fluidized state with a stream of non-reducing gas at elevated temperatures. One of the objects of such treatment is the removal of moisture from the catalyst, since water is a catalyst poison in many applications. Control problems are inherent in such processes because while it is desirable to maintain a steady fluidization of the catalyst bed and constant gas throughput rate, it is also necessary to remove impurities and catalyst poisons from the system continuously. Good temperature control of the catalyst bed depends upon effective fluidization and a stabilized gas-catalyst contact rate.

In their simplest form, cataylst activation processes comprise drying and heating air and passing it through a catalyst bed at a constant rate until the catalyst reaches the desired temperature, at which point the temperature of the activating air is stabilized and the catalyst is held at the activation temperature for the proper length of time. In such a process exhaust gases are vented since attempts to recycle them result in the buildup of impurities within the system and the consequent poisoning of the catalyst. Such a process is expensive because of low heat economy and the cost of a solids separation equipment which must be used to remove the catalyst fines from the exhaust stream. Such catalyst removal is essential both from the material loss standpoint and because catalytic materials are often quite corrosive or may constitute a health hazard if discharged into the atmosphere.

It is an object of this invention to provide a catalyst activation process having improved thermal efficiency. It is another object of this invention to provide a catalyst activation process in which catalyst poisons are purged from the system and makeup gas is added in such a manner that the catalyst fluidization rate can be maintained substantially constant. It is another object of this invention to provide a process in which the optimum period for catalyst activation can be determined by dew point measurements of the recirculating activation gas. It is still another object of this invention to provide an improved apparatus suitable for catalyst activation processes, and still another object is to provide a control system to be used with such an apparatus. Other objects and advantages and features will become apparent to those skilled in the art from the following description and appended drawing which depicts schematically the process and apparatus of

the invention.

According to the present invention there is provided a process for activating solid catalyst which includes contacting said catalyst with a stream of inert gas at an activation temperature and at a velocity sufficient to maintain said catalyst in a fluidized state, comprising maintaining said stream of gas in a closed recirculating system, adding make-up gas to said recirculating stream at a predetermined rate, and purging gas from said recirculating stream at a rate substantially equal to said make-up gas rate thereby enabling a circulation rate to be maintained which is substantially independent of said make-up and purge rates.

The present invention also provides apparatus for activating solid catalyst particles and in particular a particulate metallic oxide catalyst comprising an elongated activation chamber having a lower fluidizing section, an upper disengaging section having a cross-sectional area substantially greater than the cross-sectional area of said fluidizing section, a substantially frustoconical section connecting said upper and lower sections, the sides of said connecting section having a greater slope than the angle of repose of said catalyst, a grid horizontally disposed across the bottom of said lower section, means for admitting a stream of air through said grid into said lower section at a velocity sufficient to maintain said catalyst particles in a fluidized state, an outlet for said stream of air at the top of said upper section, a blower, a first conduit connecting said chamber outlet and the intake of said blower, a purge line leading from said first conduit, means for filtering and drying air, a make-up line leading to said first conduit from said filtering and drying means for the introduction of make-up air at a rate equal to the rate at which gas is purged, air heating means, and a second conduit connecting the exhaust of said blower and said chamber inlet and passing through said air heating means.

Still another feature of the invention is a control system which monitors the rate of fluidizing air by detecting the pressure drop across the fluidized catalyst bed, in cooperation with a rate of flow controller in the gas makeup line and a pressure controller which balances the purge rate with the makeup rate. An additional feature of the invention is a dew point indicator which enables an accurate control of catalyst activation time.

In its broadest aspect the invention deals generally with the activation of catalyst by controlled heat treatment while in a fluidized state, and many different types of catalytic materials can be advantageously treated by the process of the invention. It is further practicable to utilize the apparatus of the invention in catalyst regeneration operations by varying the operating conditions to achieve the effects desired. More particularly, however, the invention deals with and was developed especially for the activation of particulate metallic oxide catalytic materials such as those used in the polymerization art. A catalyst for which the invention is especially valuable is a recently developed polymerization catalyst comprising chromium oxide, a portion of which is hexavalent (preferably at least 0.1 per cent by weight of the total catalyst). In this catalyst the chromium oxide is associated with silica, alumina, thoria, or zirconia, or a mixture of these oxides; and the total chromium content is preferably between 0.1 and 10 weight per cent. This catalyst is extremely valuable in the polymerization and copolymerization of polymerizable olefins, especially aliphatic and cyclic olefins including both mono- and diolefins, for example, ethylene, and butadiene. Among the examples of the preferred class of products which can be obtained in processes using this catalyst are homopolymers of ethylene, propylene, 1-butene, and 1-pentene, and copolymers of ethylene with propylene, 1-butene, 2-butene, or butadiene. A preferred use for this catalyst is the polymerization of aliphatic 1-olefins with a maximum of 8 carbon atoms per molecule and no branching nearer the double bond than the 4 position. Such a polymerization is ordinarily carried out at a temperature between 150 and 450°F., and the pressure can vary over a wide range, for example, from atmospheric to 1000 pounds per square inch absolute or above. It is preferred that a solvent be used which is liquid and inert under contacting conditions, such as naphthenic hydrocarbons and paraffinic hydrocarbons of from 3 to 12 carbon atoms, for example, isooctane and cyclohexane. In such cases the catalyst can be employed in a slurry form in the solvent contacting the polymerizable material which is dissolved in the solvent although fixed bed systems are also operable.

The activity of such a catalyst is considerably affected by the manner in which it is made and treated prior to the polymerization step. A catalyst of the type described can be prepared by impregnation of a synthetic silica-alumina gel cracking catalyst with chromic acid. The catalyst base is immersed in an aqueous solution of chromic acid of proper concentration to produce a catalyst with the specified chromium content. Following the impregnation step the catalyst is allowed to drain until the excess liquid is removed. The catalyst is then dried at a relatively low temperature, for example about 150°F. and below, to a water content of about 3 to 5 weight per cent, after which grinding and classification may be necessary to produce a catalyst having an average particle size in the desired range. Catalysts ranging in average particle size from 10 to 500

microns can be satisfactorily activated using the invention. The raw catalyst is then activated by fluidizing it in air and heating it while fluidized to an elevated temperature. It is ordinarily maintained at this elevated temperature for a length of time depending upon the temperature used and degree of activation desired, and then cooled and stored in an inert atmosphere prior to being used for polymerization.

The process variables for catalyst activation can vary over a wide range and the catalyst will still exhibit some activity. There are, however, optimum conditions for each type of catalyst, and the invention provides the process and apparatus by which these variables can be effectively controlled.

One embodiment of the invention includes a closed gas circulating system comprising a blower, an activation chamber, and conduits connecting the exhaust of the blower with the bottom of the chamber and the top of the chamber with the intake of blower. Downstream of the blower is a heater which heats the gas on its way to the activation chamber, and downstream of the chamber is a cooler which cools the gas before it re-enters the blower. Also downstream of the activation chamber is a purge outlet which should include suitable filtering means, such as fiber, ceramic, or metal filters to remove catalyst fines. If bag filters are used it is preferred that the purge outlet be located downstream from the cooling means. Upstream from the blower a makeup gas inlet is located to replace gas purged. This gas should be filtered and cooled to reduce the load on the recirculating gas cooler and part of this makeup gas can be introduced at the blower bearings to serve as cooling media for these bearings.

Solid, granular catalyst having an average particle size in the range of 10 to 500 microns is charged to the activation chamber forming a bed therein on a grid or perforated plate. This catalyst bed is fluidized by passing a stream of air through it at a velocity less than the terminal velocity of most of the catalyst particles. Some of the catalyst will be entrained in the air stream, however, and in the upper portion of the activating chamber the cross-section is increased to lower the air velocity and drop out these catalyst particles. The air leaving the activation chamber can be passed through a cyclone separator to remove residual catalyst from the stream although it is quite probable that even so, some catalyst fines will remain in the recirculating stream and be collected in the purge filters. Normally the catalyst bed will be fluidized during charging and after the batch is in the activator the heating of the circulating air is begun. When activation temperature is reached in the catalyst bed the temperature is stabilized and held for a period of time and then cooled. The activated catalyst is discharged from the activation chamber and a new batch is charged to repeat the cycle.

To explain the invention more clearly, reference is now made to the attached drawing. In this drawing an elongated vessel 11 is the catalyst activator. This vessel comprises a lower section 12 in which the catalyst fluidization occurs and an upper section 13 which serves to disengage the catalyst particles from the fluidizing air stream by reducing the velocity thereof. It is necessary, therefore, that the upper disengaging section have a larger cross-section than the lower fluidizing section. The ratios of cross-sectional area are to a certain extent a problem in economics, and ratios as high as 40 to 1 are theoretically but not economically practical. It is preferred, therefore, that the disengaging section have a cross-section of about four times that of the fluidizing section. These two sections of the activator are connected by a third section 14 which is substantially frustoconical, having sides with a greater slope than the angle of repose of the catalyst. For most materials contemplated, a slope 60° would be adequate. At the lower end of the fluidizing section a grid 15 or perforated plate is horizontally disposed, having a relatively low percentage of free area, for example about 1 to 5 per cent with holes of approximately 0.25 to 3 inches in diameter, depending on the catalyst particle size. These figures are exemplary and the optimum free area will also depend upon the over-all size of the activation vessel. Near the lower part of the disengaging section an inlet 16 is provided for the introduction of granular catalytic materials which, when introduced to the activator, form a bed upon grid 15 in the fluidizing section. Below grid 15 inlet means 17 is provided for the introduction of the activating air stream and an outlet 18 for the stream is provided at the top of the disengaging section. The outlet is also equipped with a cyclone separator 19 which separates catalyst particles not removed from the air stream in the disengaging section. A conduit 21 connects the cyclone with blowers 22 and 22a in series, and conduit 23 connects the exhaust of the blowers with activator chamber inlet 17, thus completing the recycle circuit. Extending from conduit 21 is purge line 24 leading to bag filters 25; and leading into line 21, downstream of purge line 24, is makeup line 26 through which fresh activating gas is introduced to the system. Air filter 27, blower 28, condenser 29 and knockout drum 31 are connected in series to filter and partially dry the makeup gas, which is usually air. Conduit 32 connects the knockout drum with air dryer 33, which in turn is connected by conduit 34 to a second dryer 35. Any suitable desiccant, such as bauxite,

can be used in these dryers which are of conventional design. Also a regeneration heater can be used in combination with this system to heat a portion of the air from conduit 32 and pass it through a dryer not in use, thereby regenerating it. Bypass lines 36 and 37 enable the bypass of either or both dryers.

In conduit 21, upstream from the purge outlet line 24, is an air fin heat exchanger 38 coupled with a bypass line 39. These features enable the activation air to be cooled prior to passing through the bag filters 25 or the blowers 22. Heat exchanger 38 is also used to cool the recirculating air stream during the cooling portion of the activation cycle. The air stream is reheated in conduit 23 by air heater 41 prior to reentering the activation chamber. Air heater 41 can be of any suitable type, for example, a tubular gas-fired heater

The temperature of the air stream in conduit 21 is controlled by temperature controller 42 having a temperature sensitive element 43 in conduit 21 and operatively connected to control valve 44 in cooler bypass 39. In processes where higher activation temperatures are used, for example above 600°F., it is desirable to partially cool the recycle stream so that expensive blowers and catalyst separation equipment will not be required.

To enable a satisfactory control of a substantially constant air velocity for fluidization, differential pressure controller 45 is used in combination with pressure lines 46 and 47 positioned at upper and lower points, respectively in the fluidizing section to measure the pressure drop therethrough. Differential pressure controller 45 is operatively connected to control valve 48 in conduit 23, thereby regulating the rate at which the air stream enters the activator. Turbine control valves 49 and 49a are also regulated by pressure controller 45, thus varying the speed of blowers 22 and 22a. This combination enables major air velocity changes to be made by regulating the speed of the blowers while more sensitive adjustments can be made with valve 48. Makeup air in line 26 is controlled by flow recorder 51 controlling valve 52 and thereby enables a constant addition of makeup air to the system. Pressure controller 53 controls valve 54 in purge line 24 and serves to stabilize the pressure within the system by purging an amount of air approximately equal to the air added through line 26.

In the process of this invention a catalyst activation is carried out batchwise. Raw catalyst in hopper 55 is conveyed by a pneumatic system comprising line 56 and blower 57 through valve 58 into activator 11. Catalyst falls into the fluidizing zone 12, forming a bed depth which is satisfactory for fluidization. For example, when a catalytic material of the type and size described above is fluidized, the optimum bed depth is approximately 4 feet. A minimum bed depth is preferred for any given quantity of catalyst to allow a greater cross-section and hence greater gas volume flow rate which in turn reduces activation cycle time. The fluidizing section in such a case should be 8 to 10 feet long to allow for approximately 100 per cent bed expansion under fluidizing conditions. After the activator has been charged, valve 58 is closed and pump 57 is shut off. The bed is fluidized by passing an air stream from blowers 22 through line 23 into inlet 17. The fluidizing velocity depends upon catalyst particle size. The minimum velocity must be sufficient to cause incipient fluidization, and the maximum is the terminal velocity of the catalyst particles. Ordinarily with catalyst of the size described the air velocity in the fluidizing zone is about 0.1 to 10 feet per second and preferably from about 1.2 to 1.6 feet per second. In the disengaging zone the air velocity is reduced considerably to permit entrained catalyst particles to return to the fluidizing section. In this section the velocity is ordinarily from about 0.01 to 9 feet per second and preferably from about 0.3 to 0.4 feet per second. This would allow a satisfactory disengaging of almost all of the catalyst before it leaves the activator. The air is recirculated to the blowers and heated to bring the catalyst to the satisfactory activation temperature. Meanwhile, fresh air is brought into the system through makeup line 26, having been filtered, cooled, and the moisture removed. During the initial part of the cycle the makeup air bypasses the dryers 33 and 35 and is added at a constant rate equal to a small fraction of the total recirculation rate, for example, about 5 to 25 volume per cent, preferably about 10 per cent. Adding the makeup air to the system causes the pressure to increase, which is offset by purging an approximately equal amount of air through line 24. In this way moisture and catalyst poisons are purged from the system and the catalyst is activated with a recirculating stream that is substantially independent of the makeup and purge rates. Also, during the "heat-up" portion of the activation cycle the pressure of the system tends to increase and the purge rate is thereby greater. This is advantageous because during this part of the cycle the amount of moisture being driven off the catalyst is highest and hence the need for purge is greater. Likewise, during the cooling part of the cycle the purge rate is lower and the need for purge is less since the moisture has by this time been removed.

Although it is possible to use undried air throughout the entire activation cycle, the activity of the catalyst suffers accordingly. It is not necessary, however, to dry the air at the beginning of the cycle. Undried air can

be used until the catalyst temperature nears the desired activation temperature at which point the makeup air is passed through the dryers. The activation temperature can vary over a broad range, for example, from 400 to 1750°F., and the holding time will vary accordingly. At the highest temperature the activation time could be a matter of a few seconds, and at the lowest temperature the activation time could be as long as 40 hours or more. A reasonable activation time, however, should be less than 10 hours, and it is preferred that the holding time be about in the magnitude of 5 to 6 hours. The exact time for activation depends ultimately on variations in plant design, and it is better to control the holding period by the dew point of the activation air leaving the activator. This dew point can be measured at any point in the system if proper corrections are made for the makeup and purge streams. When possible, the dew point can be measured upstream from the point at which the makeup air enters the recycle stream, although in the embodiment shown in the drawing, dew point indicator recorder 59 is located across the blowers 22 and 22a in order to obtain maximum pressure drop for its operation. A suitable instrument of this type is a "Foxboro" Dewcel (registered trademark) plus a "Foxboro" "Dynalog" (registered trademark) recorder. These units are described on pages 33 and 64 respectively of Bulletin 450 of the Foxboro Company, Foxboro, Massachusetts. It is preferred that the catalyst be activated at a temperature in the range of 800 to 1700°F. and held at this temperature until the air leaving the catalyst mass has obtained a dew point of not over 0°F. and preferably about −40°F. or less. When an activation temperature in this preferred range is employed, the makeup air should be channeled through the air dryers when the catalyst mass has reached a temperature of approximately 750°F. After the catalyst has been activated, fuel to the air heater is cut off and the catalyst mass is allowed to cool while continuing the recirculation and fluidization. The dew point measuring instrument can be adapted to close the valve in the air heater fuel line at any predetermined dew point value. When the catalyst is cooled to approximately 100 to 200°F., it is dropped through lines 61 or 61a to catalyst receivers 62 and 62a. Interstitial air is purged from the catalyst voids by inert gas, such as nitrogen, entering through conduit 63; and an inert gas blanket is maintained over the catalyst until it is ready to be used for the polymerization process.

In further clarification of the invention, a specific example will be discussed in connection therewith.

Approximately 2,000 pounds of polymerization catalyst (dry basis) containing 2.5 weight per cent chromium oxide is impregnated on a silica-aluma base and having an average particle size of 70 microns (70% between 50 and 90 microns) is charged to the activation chamber by a pneumatic system. The raw catalyst as charged contains 200 pounds of water. During charging, air is circulated through the activation chamber to fluidize the catalyst bed.

After charging, fuel gas is fed to the heater and the temperature of the catalyst is raised to 1000°F. over a period of 6 hours. Makeup air is meanwhile fed continuously to the activation system after being filtered, cooled, and separated from condensed moisture. The makeup air flow rate is maintained substantially constant at about 10 volume per cent of the recirculation stream. When the catalyst temperature reaches 750°F., the makeup air is routed through the bauxite dryers and a dew point of −40°F. is maintained for all makeup air throughout the remainder of the activation cycle.

Recirculation air flows unrestricted through the air fin cooler bypass until the temperature of the air at the blower intake reaches 600°F., at which point the temperature controller begins operation, routing more air through the cooler in order to maintain the blower inlet air below 600°F. The air immediately downstream of the air fin cooler is about 400°F. and at this point air is purged to the bag filters thus maintaining the pressure at the purge outlet substantially constant. For satisfactory fluidization and disengaging of catalyst in the activation chamber the air velocity in the fluidizing section is about 1.3 feet per second and the velocity in the disengaging section is about 0.35 feet per second.

The fluidization of the catalyst bed is controlled by maintaining a constant pressure drop across the bed by controlling the blower speed and a valve in the activator air inlet line. Suppose, for example, that during the activation cycle a channel or "blow-hole" opens up in the catalyst bed. This reduces the pressure drop across the bed and the differential pressure controller activates the motor valve in the air inlet line to reduce the air flow therethrough. This allows the bed to settle, close the channel and the pressure drop returns to its normal level.

In order to operate this type of fluidization control successfully, a substantially constant pressure and air flow rate should be maintained within the system. The invention permits this even though the activation air is recycled. As makeup air is added to the system, the pressure tends to rise and a corresponding amount of air is purged, thus returning the pressure to normal. In this way an average of about 10 volume per cent of the activation air is continuously purged thereby enabling recirculation and fluidiza-

tion control substantially independent of the makeup and purge rates. During the heat-up and cooling portions of the activation cycle more and less air, respectively, is purged which is as desired for removal of catalyst poisons.

When the catalyst reaches 1000°F., the temperature is stabilized and the activation continued at this temperature until the dew point of the air leaving the catalyst mass reaches −40°F. After about five hours this point is reached and the fuel gas to the air heater is cut off. The catalyst is cooled to 200°F. over a period of 6 hours, after which it is dropped to a catalyst receiver. Nitrogen is introduced at the bottom of the receiver and air is purged from the catalyst mass. When the air has been removed, a blanket of nitrogen is maintained over the catalyst until it is needed for the polymerization reaction. About 1900 pounds of activated catalyst are produced and about 100 pounds of catalyst fines are recovered from the bag filters. Substantially all of the 200 pounds of water is removed.

While this invention has been discussed in terms of specific examples and embodiments, it should be understood that these examples have been used for illustrative purposes only, and several modifications can be made within the scope of the invention by those skilled in the art.

WHAT WE CLAIM IS:—

1. A process for activating solid catalyst particles which includes contacting said catalyst with a stream of inert gas at an activation temperature and at a velocity sufficient to maintain said catalyst in a fluidized state, comprising maintaining said stream of gas in a closed recirculating system, adding make-up gas to said recirculating stream at a predetermined rate, and purging gas from said recirculating stream at a rate substantially equal to said make-up gas rate thereby enabling a circulation rate to be maintained which is substantially independent of said make-up and purge rates.

2. A process according to claim 1, wherein said make-up gas is dried before being added to said circulating stream, the catalyst being heated to substantially activation temperature before commencing said drying step.

3. A process according to claim 1 or 2, comprising introducing the catalyst to a fluidizing zone and forming a bed therein, passing said circulating gas stream, which is preferably a non-reducing gas such as air, through said bed at a velocity sufficient to fluidize said bed, heating said catalyst to activation temperature and maintaining said temperature for a predetermined period of time, disengaging catalyst from said gas stream in a disengaging zone by reducing its velocity therein, withdrawing said gas stream from the disengaging zone and recirculating said stream to the fluidizing zone.

4. A process according to claim 3, wherein said recirculating stream is heated before entering the fluidizing zone.

5. A process according to claim 4, wherein at least a portion of said recirculating stream is cooled prior to being heated.

6. A process according to any one of the preceding claims, wherein the catalyst bed and gas stream passing therethrough are cooled after said catalyst has been held at activation temperature for a time sufficient to activate same.

7. A process according to any one of claims 3-6, wherein a substantially constant pressure drop is maintained across the catalyst bed by controlling the rate of flow of said gas stream to the fluidizing zone responsive to changes in said pressure drop.

8. A process according to any one of claims 3-7, wherein the period of time said catalyst is held at activation temperature is controlled according to the dew point of said circulating gas stream.

9. A process according to any one of the preceding claims, wherein said catalyst comprises chromium oxide catalyst particles, a portion of said chromium being in the hexavalent form.

10. A process according to claim 9, wherein the catalyst is heated to a temperature in the range of 400° to 1750°F. for a predetermined period of time.

11. A process according to claim 9 or 10, wherein said catalyst is heated to substantially 750°F. before beginning to dry the make-up air, and the activation temperature is maintained in the range of 800° to 1700°F.

12. A process according to any one of claims 3-11, wherein the dew point of the circulating air is measured at a point upstream from the point at which the make-up air enters the recycle stream and said stream is cooled when the dew point drops to 0°F. or below.

13. A process according to claim 1, wherein said catalyst comprises chromium oxide, a portion of which is hexavalent, said catalyst having an average particle size in the range of 10 to 500 microns, comprising forming a bed of said catalyst in an activation zone, fluidizing said catalyst bed by passing a circulating stream of air therethrough at a velocity in the range of 0.1 to 10 feet per second, disengaging catalyst carry-over suspended in said air stream by reducing the velocity thereof within the activation zone, maintaining the pressure in said circulating stream of air substantially constant by purging air from said circulating stream downstream of said activation zone and adding make-up air to said circulating stream at an approximately constant rate, heating said recirculating air so that said catalyst is heated to substantially 750°F. and

thereafter drying said make-up air, continuing to heat said circulating air until said catalyst reaches an activation temperature in the range of 800 to 1700°F., holding said activation temperature until the dew point of said circulating air leaving said catalyst bed reaches a value in the range of about −40°F. and below, and thereafter cooling the recirculating air and catalyst, said purging and make-up rates being such that the velocity of said circulating air stream is substantially independent of the make-up and purge rates and temperature changes within the system.

14. A process according to claim 13, wherein said disengaging velocity is in the range of 0.01 to 9 feet per second.

15. A process according to claim 13, comprising passing said circulating air stream through the catalyst bed at an air velocity in the range of 1.2 to 1.6 feet per second; disengaging catalyst from said air stream within said activation zone by reducing the air velocity to 0.3 to 0.4 feet per second; adding make-up air to said recirculating stream at a substantially constant rate equal in amount to 5 to 25 volume per cent of said circulating air stream; holding said activation temperature for 5 to 6 hours until the dew point of the circulating gas from the activation zone is in the range of substantially −40°F. and below; thereafter cooling said circulating stream and catalyst to 100-200°F.; purging air from said circulating stream at a point downstream from said activation zone in an amount necessary to maintain a substantially constant pressure at said downstream purge point thereby maintaining a substantially constant volume of air in said recirculating air stream during said heating, holding, and cooling steps; discharging said catalyst from said activation zone; and blanketing said catalyst thus activated with inert gas.

16. Apparatus for activating solid catalyst particles and in particular a particulate metallic oxide catalyst, comprising an elongated activation chamber having a lower fluidizing section, an upper disengaging section having a cross-sectional area substantially greater than the cross-sectional area of said fluidizing section, a substantially frustoconical section connecting said upper and lower sections, the sides of said connecting section having a greater slope than the angle of repose of said catalyst, a grid horizontally disposed across the bottom of said lower section, means for admitting a stream of air through said grid into said lower section at a velocity sufficient to maintain said catalyst particles in a fluidized state, an outlet for said stream of air at the top of said upper section, a blower, a first conduit connecting said chamber outlet and the intake of said blower, a purge line leading from said first conduit, means for filtering and drying air, a make-up line leading to said first conduit from said filtering and drying means, air heating means, and a second conduit connecting the exhaust of said blower and said chamber inlet and passing through said air heating means.

17. Apparatus according to claim 16, wherein said activation chamber is cylindrically shaped, said upper disengaging section having a diameter about twice the diameter of said fluidizing section.

18. Apparatus according to claim 17, comprising means for measuring the pressure drop across said fluidizing section and producing an output proportional thereto, separate means for controlling said blower operation and air stream flow through said second conduit in response to said output, thereby enabling automatic control of substantially constant air flow rates in said fluidizing section, a rate of flow regulator in said make-up line, and a pressure flow controller in said purge line, thus enabling control of purge rates thereby to permit fluidizing rates substantially independent of the purge and make-up rates and temperature changes within the system.

19. Apparatus according to claim 17 or 18, comprising a dew point measuring apparatus adapted to measure the dew point of air coming from said activation chamber and extinguish said air heating means when said dew point reaches a predetermined value.

20. A process substantially as hereinbefore described with reference to the Example.

21. A process substantially as hereinbefore described with reference to the accompanying drawings.

22. Apparatus substantially as hereinbefore described with reference to the drawings.

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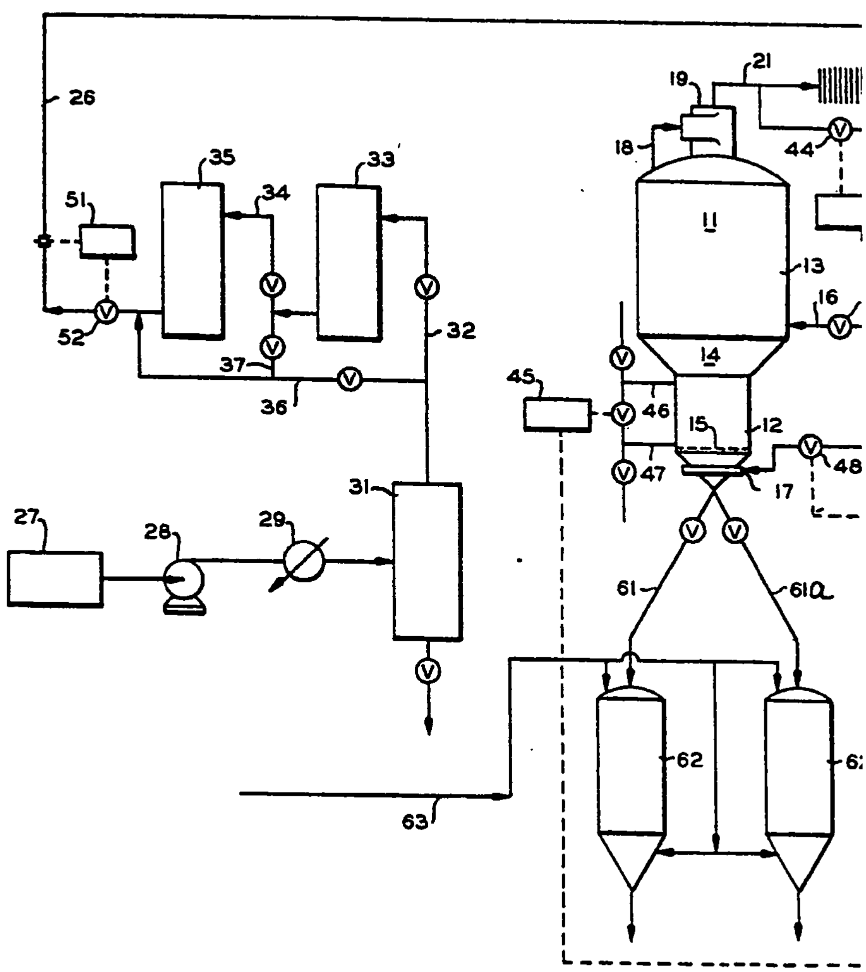

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849,929 COMPLETE SPECIFICATION

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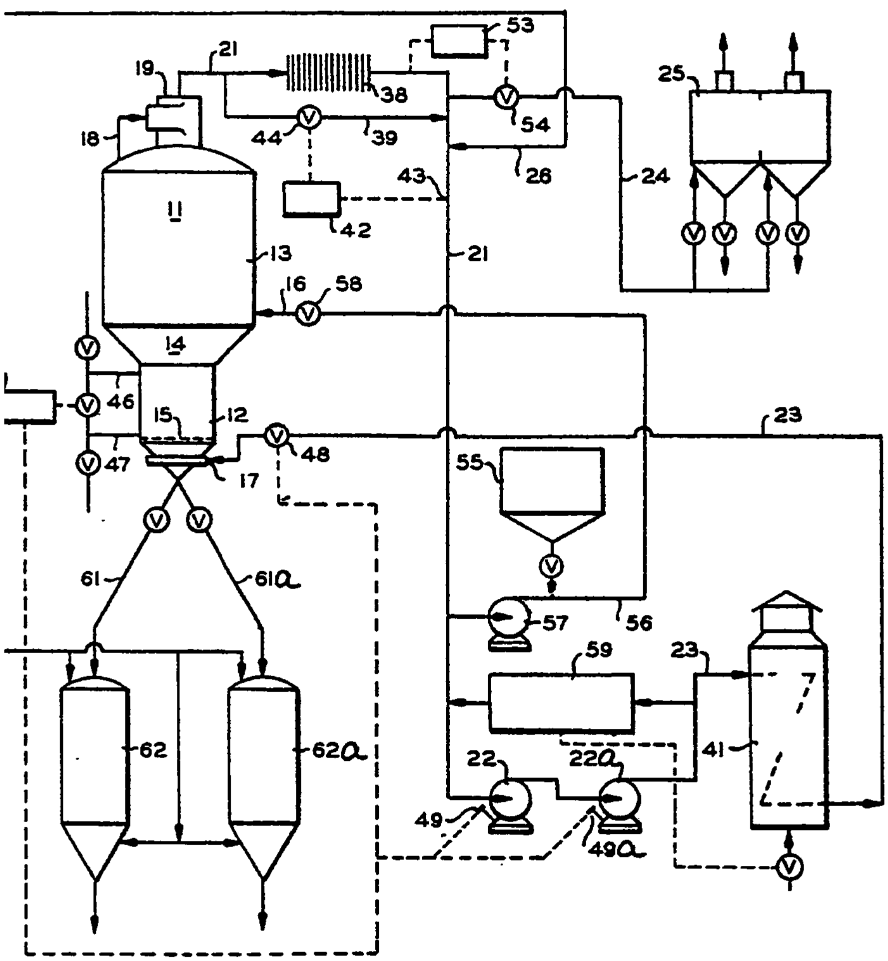

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